



STUDY OF TECHNOLOGY OF MECHANICAL ACTIVATION OF MONTMORILLONITE

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Abstract. The work consists of studying the physicochemical properties of natural (mine) bentonite mineral raw materials by the method of mechanical activation. X-ray phase and X-ray spectral analyses of the layered composition of montmorillonite were carried out. Mechanochemical activation of zeolites to change the surface area and particle size was carried out using a powerful AGO-3 mill and a VKMD-6 conical vibration mill. It was proven that mechanical activation of bedded montmorillonite depends on the time of 20, 40, 80 and 160 s, increasing its surface area. Optimum modes were determined by doubling the time of mechanical activation and studying the density, sorption properties and specific surface of the zeolite using the Karman-Kozeny method.

Keywords: Bentonite, montmorillonite, X-ray phase, X-ray spectral, mill, conical vibratory mill, Karman-Kozeny, surface area.

Introduction. Currently, there is a significant increase in interest in the creation of new environmentally friendly sorbents, fixatives and catalysts based on natural clay materials and aluminosilicates. Natural zeolites are a unique mineral with catalytic, adsorption and ion-exchange properties and are a relatively inexpensive raw material compared to synthetic materials. Currently, about two hundred different types of zeolites are used; they are divided into inorganic, organic and synthetic types. Due to the favorable arrangement of absorbed molecules, zeolites have well-developed mesoporous crystals, which are molecular sieves [1,2]. Their crystal lattice is formed in the form of a silicon-oxygen tetrahedron consisting of four, five or more rings. In some of these tetrahedra, the silicon atom is replaced by aluminum. The negative charges of the resulting crystal lattice nodes are replaced mainly by sodium, calcium and potassium cations. Such a structure of water molecules are located among the mesoporous cations bound together in the internal crystalline space of zeolites [12-16]. The porous mesostructure of zeolites has the best properties: adsorption, molecular sieve, ion exchange and catalytic properties.

(Navahor district, Navoi region) studied its physicochemical properties by the method of mechanical activation of natural (mine) bentonite mineral raw materials [17-20].

Research object Navbahor deposit of bentonite clays - Navbahor district of Navoi region, Jan. On the southwestern slope of the Nurotinsky ridge. It is located 12 km north of the village of Kalkanota. In 1998, A. U. Mirzaev and H. Opened by Chinikulov. The Navbahor district bentonite deposit contains large reserves of natural montmorillonite, which are extracted from open layers. Currently, there is a significant increase in interest in the creation of new environmentally friendly sorbents, fixatives and catalysts based on natural clay materials and aluminosilicates. Natural zeolites are a unique mineral with catalytic, adsorption and ion-exchange properties and are a relatively inexpensive raw material compared to synthetic materials. Currently, about two hundred different types of zeolites are used; they are divided into inorganic, organic

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Experimental part Montmorillonite is the most common natural zeolite, its crystals consist of a three-dimensional aluminosilicate-oxygen tetrahedron $(Si,Al)O_4$, it is a mineral with a large surface area, consisting of micro-, meso- and macropores.



Figure 1. Montmorillonite and protrusions of the montmorillonite structure.

Chemical formula: $(Na,Ca)_{0,3}(Al,Mg)_2[Si_4O_{10}](OH)_2 \cdot nH_2O$ [1,3]. In natural montmorillonite, the molar ratio of SiO_2 / Al_2O_3 is 9-12 or more [4]. Montmorillonite, which belongs to the group of thin-plate zeolites, is very resistant to dehydration and adsorbs H_2O and CO_2 well [5]. A schematic model of montmorillonite and the structure of montmorillonite is shown in Figure 1 [6-10]. At the same time, the thermal stability of montmorillonite in air is often about 550-600 °C, and the pore diameter of montmorillonite is 0.2 nm [1, 11].

Element	Mass fraction, %
Si	33,57
O	40,08
Mg	0,6
Al	6,59
C	6,80
K	1,1
Ca	8,73
Fe	2,53

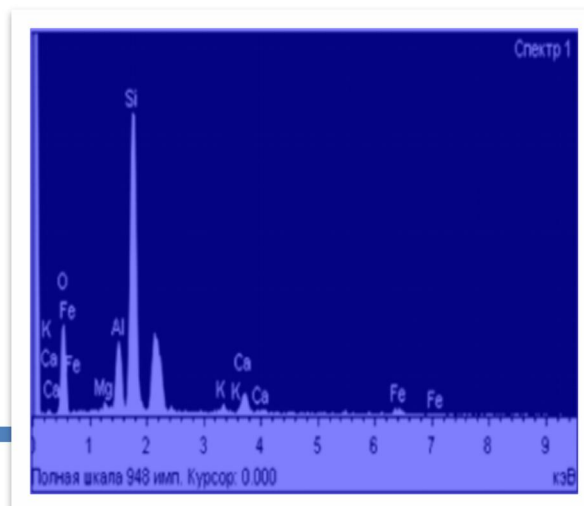
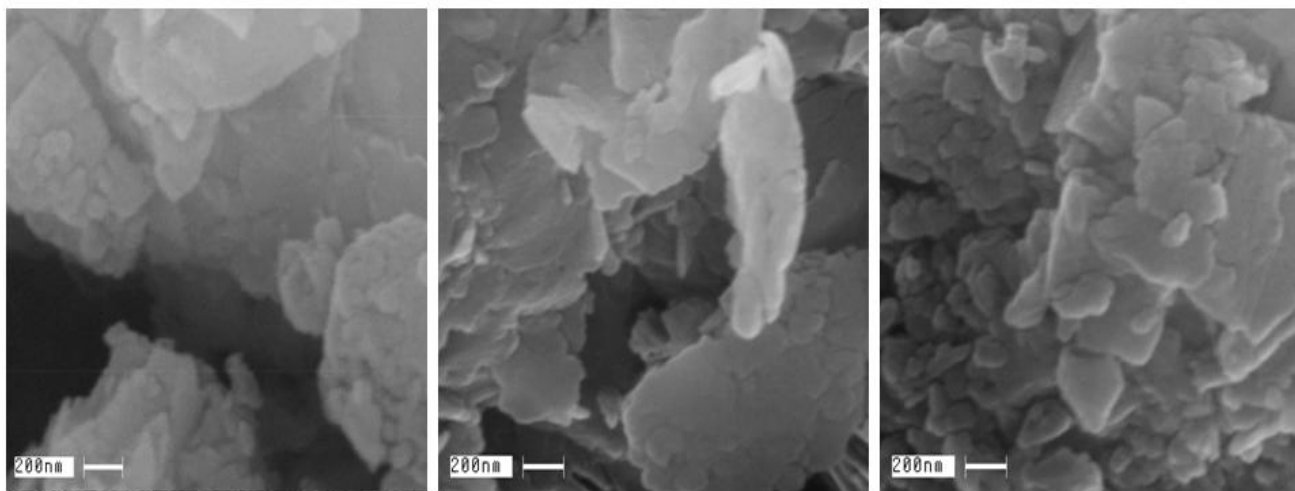


Figure 1. Elemental composition and X-ray spectral analysis of the original montmorillonite from the formation.

Mechanochemical activation of zeolites to change their surface area and particle size was performed using a powerful AGO-3 mill and a VKMD-6 conical vibration mill. Grinding in the AGO-3 mill was carried out at intervals of 40, 80 and 160 s, doubling the grinding time. Grinding in the VKMD-6 was carried out in ascending order of number (1,2,4,8). Determination of the specific surface of zeolites by the Karman-Kozeny method showed that with an increase in the mechanical activation time and the number of grinds, the specific surface of the particles quickly increases, and the maximum time of 160 s corresponds to mode "8" in the AGO-3 and VKMD-6, mechanical activation is performed.

Experimental results and their discussion

The surfaces of the obtained fine particles of montmorillonite powder were examined by electron microscopy (Fig. 2, 3). An electron photograph of the montmorillonite surface after 40, 80 and 160 seconds of mechanical activation in the AGO-3 mill is shown.

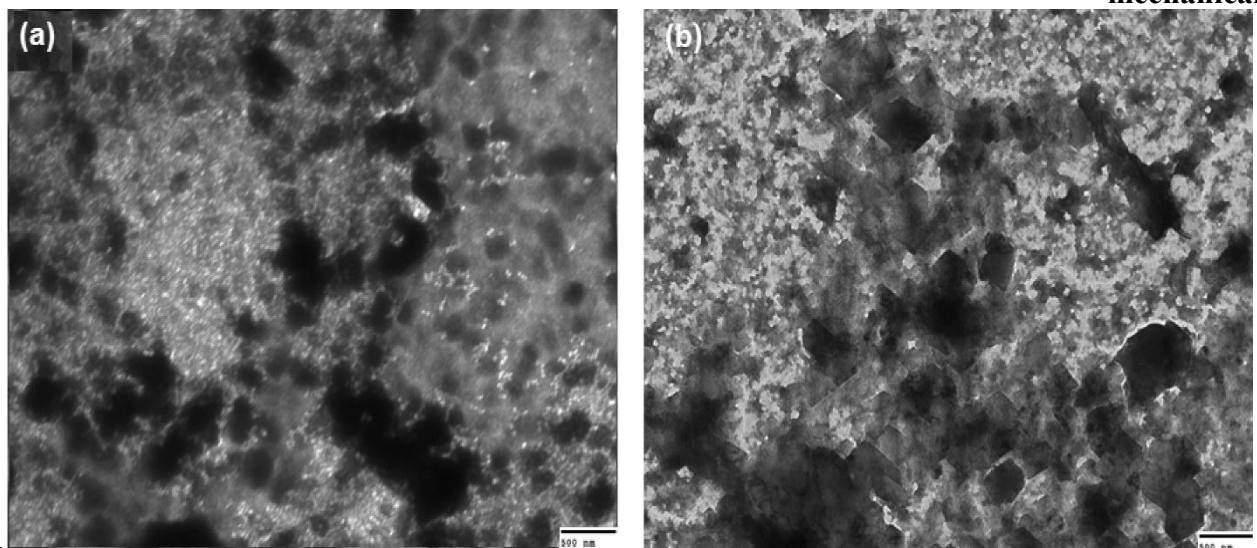


МА - 40 секунд

МА - 80 секунд

МА - 160 секунд

2 - picture In the sample of zeolite AGO-3 surfaces a) 40 s; b) 80 s; c) 160 s; SEM photograph in case of mechanical



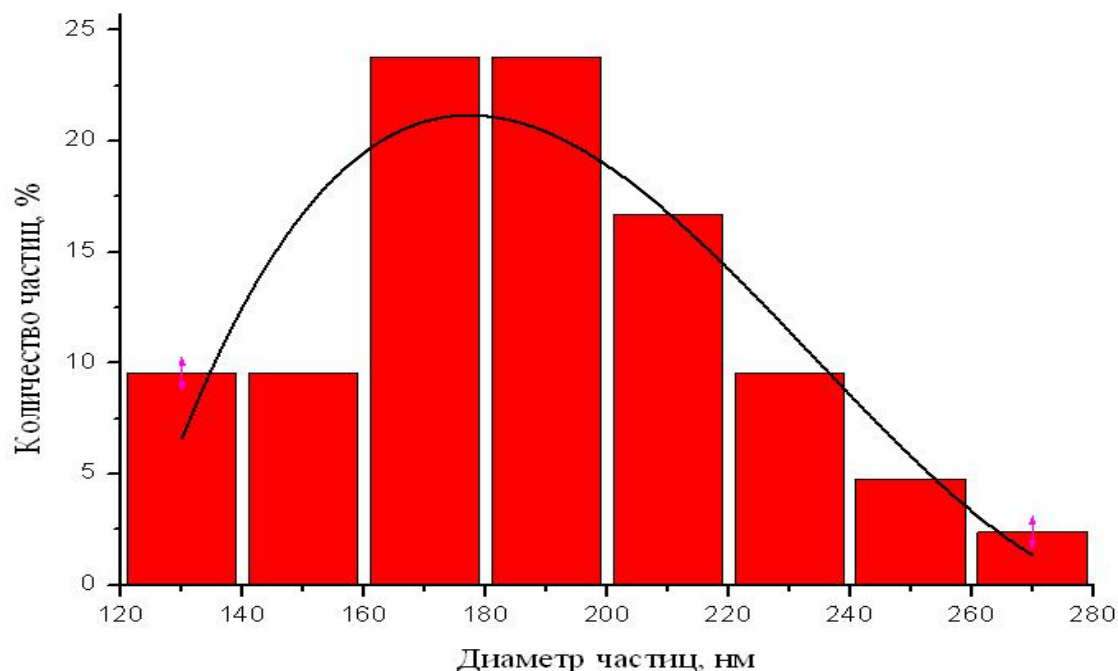
activation

3 – Fig. Zeolite Dispersion "8" in VKMD-6 above-water in the mode: a) initial sample b) from grinding the next sample SEM photographs

Optimum conditions were determined by doubling the mechanical activation time and studying the density, gas permeability and specific surface area of the zeolite using the Karman-Kozeny method. As the

mechanical activation time increases, the relative particle size in the powder decreases. Granulometric analysis of the zeolite was performed to obtain information on the granulometric composition. It can be seen that with increasing grinding of montmorillonite, the size of its particles decreases.

VKMD-6 Fig. 4 shows the values of the measured quantities, the equivalent particle diameter and the surface morphology of the zeolite particles depending on the amount of



dispersion.

Figure 4. Particle size distribution

From the data in Figure 4 it is evident that the value of the specific surface area of the zeolite particles changes depending on the time of mechanical activation. As the time of mechanical activation increases, the specific surface area of the particles increases rapidly, and after reaching a maximum at 160 s, the activation begins to decrease.

Table 1.

Dependence of the specific surface area of AGO-3 for layered montmorillonite on the time of mechanical activation.

No	20 seconds	40 seconds	80 seconds	160 seconds
cm ² /g	cm ² /g	cm ² /g	cm ² /g	cm ² /g
1	33 6	92 9	1770	2357
2	33 6	129 1	2500	2421
3	33 6	131 4	1740	2326
4	33 6	1204	1802 г.	2356
5	33 6	900	1802 г.	2357
6	35 2	86 6	1772 г.	2356
7	33 6	86 6	1843 г.	2326
8	33 6	875	1772 г.	2356
9	35 2	89 1	1772 г.	2357
10	33 6	89 1	1773 г.	2357
Average cost	34 1	1003	1855 г.	2357

The sorption properties are affected by such factors as the degree of crystallinity. This property was determined for mechanically activated samples. The values obtained for the mechanically activated sample using the following formula are presented in Table 2.

$$k = \frac{(\sum I_{i1})}{(\sum I_{i2})} * 100\%$$

k - degree of crystallinity, %;

I₁ – highest intensity of 1st initial test sample;

I₂ – peak intensity of mechanically activated sample.

Table 2

Degree of crystallization of mechanically activated powder samples

Zeolite property	Degree of crystallinity, %
Mode "6" in VKMD-8	90
AGO-3 in mechanical activation mode 160 p.	94,5

It was found that the samples differ in the degree of crystallinity, and the maximum mode of 160 s in AGO-3 corresponds to a higher value of this coefficient compared to the mode "6" in VKMD-8.

It can be said that it has been experimentally established that the specific surface and the degree of crystallinity are directly proportional to each other, therefore, the larger the surface area, the higher the degree of crystallinity.

Bentonites are not considered pure raw materials and, in addition to the main phase of montmorillonite, contain a mixture of various minerals, depending on the deposit from which they are mined. To determine the mineralogical composition of bentonites, X-ray phase analysis was carried out. Fig. 5 shows X-ray diffraction patterns of natural bentonites, as well as bentonites calcined in an inert argon atmosphere at 550 °C

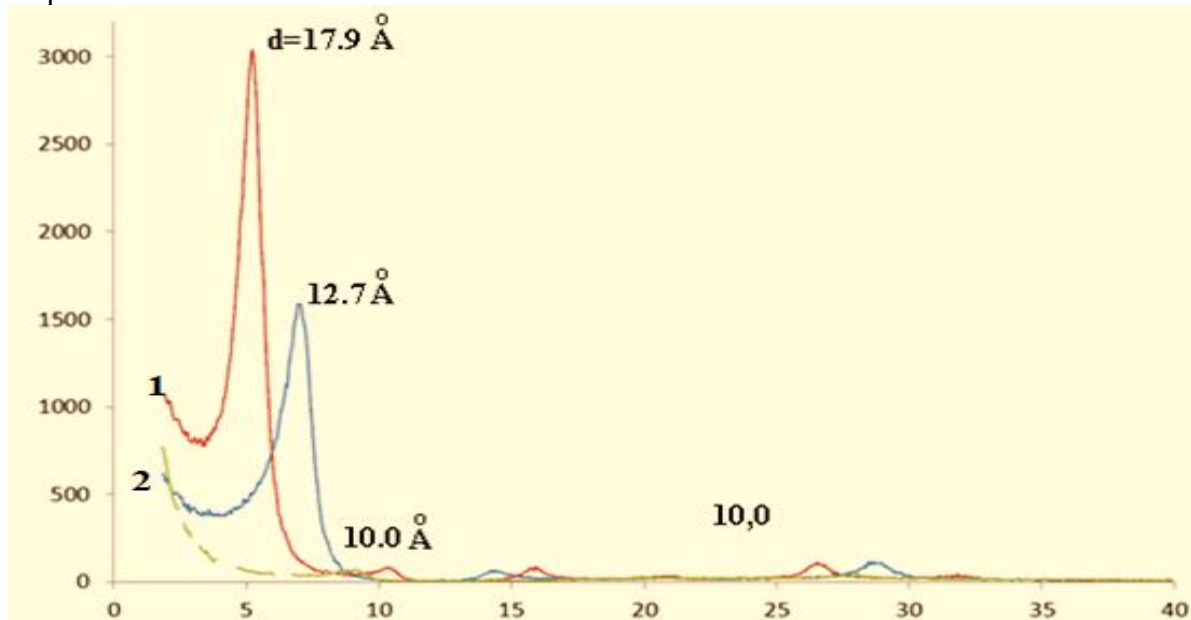


Fig. 5. Diffraction patterns of natural Navbahor bentonite (1) and a sample calcined in an inert argon atmosphere at 550°C (2) X-ray structural analysis of natural bentonites indicates the presence of montmorillonite and α-cristobalite. Annealing at 550oC for activation showed a shift of bentonites from 17.9 Å to 10 Å due to the loss of water between these layers. X-ray diffraction peaks of the studied montmorillonite powders were obtained experimentally using the mechanical activation mode.

Conclusion. The presented dependences experimentally indicate the existence of an optimal time and mode of mechanical activation corresponding to the maximum specific surface area and maximum sorption capacity of montmorillonite powders. This indicates that mechanical activation of montmorillonite is a method that improves its adsorption properties.

During mechanical movements in the AGO-3 mill, the reflection lines from the planes shown in the work disappear on the X-ray diffraction pattern for 320 seconds, which is due to the dispersion of associated particles to a nanosized state, in which X-rays occur in a large angular range. The dependence of the adsorption properties of zeolites on the degree of crystallinity is determined. For samples with a high degree of crystallinity, the adsorption properties are higher over time than for samples with a low degree of

crystallinity. In the AGO-3 planetary mill, mechanical activation is an effective grinding method and a way to improve the sorption properties of montmorillonite. X-ray structural analysis of natural bentonites indicates the presence of montmorillonite and α -crystalite. As a result of combustion for the purpose of activation, the mineralogical composition of bentonites changes and an illite phase appears.

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